IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s):	Juho Jumppanen, et al.	**Examiner:**	Krishnan S. Menon
Serial No.:	10/047,244	**Art Unit:**	1723
Filed:	January 14, 2002	**Docket:**	15208
For:	PROCESS FOR SEPARATING ESSENTIAL OILS FROM AN ESSENTIAL OIL-CONTAINING MATERIAL	**Dated:**	March 1, 2005

Confirmation No.: 5900

Mail Stop Appeal Brief-Patents
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

APPELLANTS' BRIEF II

Sir:

In response to the final rejection dated August 10, 2004, reconsideration of the patentability the above-identified application is respectfully requested in view of the following Brief of the Appellants.

CERTIFICATE OF MAILING UNDER 37 C.F.R. §1.8(a)

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Dated: March 1, 2005

Richard J. Danyko

REAL PARTY IN INTEREST

The application is currently assigned to Danisco A/S, a corporation of Finland.

RELATED APPEALS AND INTERFERENCES

There are no related appeals or interferences.

STATUS OF CLAIMS

Eleven (11) claims have been presented in this application, all of which are pending. Claims 1-8, 10 and 11 are under final rejection. Claim 9 is merely objected to as dependent upon a rejected base claim, but would be allowable if rewritten in independent form to include all limitations of the base claim and intervening claims.

Claims 1-8, 10 and 11, under final rejection, are the subject of this appeal.

STATUS OF AMENDMENTS

No amendments to the claims or specification were presented after final rejection.

SUMMARY OF THE INVENTION

The present invention is concerned with a process for separating essential oils from an essential oil—containing material. 1/13-15.

The most common process for obtaining essential oils is steam distillation followed by decantation of the essential oil from the steam distillate. 1/33 – 2/1. However the known processes for obtaining essential oils, which also include solvent extraction and mechanical separation (pressing), are not wholly satisfactory. First, a large volume of water and steam is required to extract the essential oils in a steam distillation, which is disadvantageous because the essential oils are usually contained only in minor quantities in the plant materials. Accordingly, the ratio of water to essential oil in the steam distillate is large, causing problems in the subsequent phase separation steps, and causing a significant amount of essential oil to remain dissolved in the aqueous phase.

In an attempt to remedy this deficiency, known processes may cool the steam distillate in the decantation vessel, which unfortunately does not entirely dispose with the above problems. A further known measure in such processes is the recycling of the aqueous phase from the decanter into the steam distillation vessel.

However, even when the aqueous phase is recycled from the decanter into the steam distillation vessel, the above problem with the residual essential oil content in said phase is not entirely overcome. On the contrary, when such a recycling technique is adopted, a substantial amount of essential oil is merely circulated through the system and never extracted as product, and moreover slows the production process down, because the water including residues of essential oils extracts less than essentially pure water.

A second major drawback associated with the use of a large steam volume is the substantial amount of energy required to generate the steam. One way of reducing the energy consumption is to use warm water, preferably from the decantation vessel. This, however, is not always possible as the requirements in the decantation step and those in the steam distillation step in this respect are quite the opposite: while in the decanter low temperatures are preferred in view of the phase separation and the residual amount of the essential oils in the aqueous phase, the steam distillation requires a high temperature recycling stream in order to consume less energy. As a result, the known processes need to compromise in either one or both steps.

In view of the above considerations, the inventors of the subject matter claimed herein have developed a process for separating essential oils from an essential oil—containing material, comprising the steps of:

(a) subjecting the essential oil—containing material to steam distillation

using a steam distillation vessel or extraction using an extraction vessel to produce a mixture containing the essential oil and at least one hydrophilic phase;

(b) contacting the mixture with a hydrophobic adsorbent in a column;

(c) recycling the hydrophilic phase leaving the column to the steam distillation vessel or extraction vessel;

(d) treating the adsorbent in the column with a solvent that is more hydrophobic than the hydrophilic phase to elute mixture or the essential oil from the adsorbent.

See independent claims 1 and 11 of the present application.

The applicants have found that this process allows for a quantitative or near quantitative adsorption of the essential oil from the steam distillate or similar mixtures to the adsorption column; then water flowing from the column to the distillation vessel is substantially free from the essential oils and thus capable of efficiently extracting (next part of) essential oils from plant material, which substantially reduces the extraction time; thus energy costs and other production costs will be diminished. See example 3 and comparative example 2; see e.g. page 14, lines 16 - 21). This improves the quality of the product (p. 14, lines 23 – 25, and p. 15, lines 1-7. The process of the present invention also makes use of a high temperature recycling stream (see claim 3) which can contribute to a lower energy consumption in the case, where the starting mixture is generated by steam distillation (see claim 2). The same advantages can also be obtained when the mixture is generated in an extraction process. A preferred manner of effecting separation is by chromatographic separation of essential oil (see claims 8 and 9). The separation according to the present invention is also faster than the separation by way of

decantation. The production of essential oils is improved in that the process is no longer limited by the rate of the phase separation and the highly efficient separation reduces the time needed to recover a given amount of material and thereby tremendously increases the overall efficiency of the process. In addition, the overall volume of steam or extracting solvent may be decreased due to the step of recycling the hydrophilic phase leaving the column to the steam distillation vessel or extraction vessel.

THE REJECTION ON APPEAL

Claims 1-8, 10, and 11 have been rejected under 35 U.S.C. §103(a) as allegedly unpatentable over JP (H6-227994) ("JP 994") in view of JP 60-115699 (JP 699), Chemical Engineer's Handbook, Perry and Green, 6th ed., 1984, pages 13-53 – 13-57("Perry") and Somekh (US 3,714,033). The Examiner contends that:

> "JP (994) discloses a process for separating essential oils comprising steam distillation (page 3, Para 0001)... to a mixture containing essential oils and water, contacting with divinyl benzene polystyrene adsorbent or activated carbon, and then desorbing the essential oils using a solvent that is more hydrophobic than the hydrophilic phase (Para 0036 – page 13 of the English translation: hydrophilic phase is water, eluting solvent is acetone)...The water (hydrophilic phase) temperature is at 60° C (page 8, page 8, para 0020) as in instant claim 3; the hydrophobic absorbent is synthetic polymer-divinyl benzene cross-linked-polystyrene, activated carbon, etc, as in instant claim 4 and 5. (page 8: 0016,0017); material is Cyprus (page 3: claim 2) as in instant claim 6; Cyprus or yellow oils (page 11:0030) as in instant claim 7; and the process is continuous as in instant claim 10 (page 11:0029).
>
> "JP (994) is silent on recycling the hydrophilic solvent, water, as in claim 1 of the instant application. Recycling of solvent in extractive and steam distillation is a common method taught in a standard textbook of Chemical Engineering, such as Chemical Engineer's Handbook, Perry and Green, 6th edition (see pages 13-53 through 13-57, and the figures)."
>
> Office Action of August 10, 2004 at pages 3 and 4.

The examiner further recognizes that JP '994 does not teach or suggest step (d) of independent claims 1 and 11, which recites

> "treating the adsorbent in the column with a solvent that is more hydrophobic than the hydrophilic phase to elute the essential oil from the adsorbent."

In addressing this deficiency of the primary reference (i.e., JP '994) the Examiner relies upon JP '699, which he alleges – erroneously -- supplies the teaching of step (d):

> "JP 994 does not teach adsorption and elution from the same column as in claim 1 step (iv) or claim 8 – chromatographic separation. JP 699 teaches *adsorbing* in to a packed column *and then eluting* the essential oils *from the same column* using a solvent more hydrophilic (pages 5 and 6 of the English translation). It would be obvious to one of ordinary skill in the art at the time of invention to sue the teaching of JP 699 in the teaching of JP 994 to elute the essential oils form the adsorption column to 'selectively' recover (by chromatographic separation) the essential oils as taught by JP 699." (Emphasis added).
>
> Office Action of August 10, 2004 at page 4.

ISSUES PRESENTED

Whether the rejection of the claims under 35 U.S.C. § 103 (a) must be reversed when, as submitted by the Applicants, the prior art does not teach an element of the invention as claimed, namely, step (d) of independent claims 1 and 11, which recites

> "treating the adsorbent in the column with a solvent that is more hydrophobic than the hydrophilic phase to elute the essential oil from the adsorbent."

The applicants submit that the answer is "yes".

Also, a second issue is whether the combined teachings of the prior art in actuality teach away from the claimed invention, in that, contrary to step (d) of independent claims 1 and 11, said combined teachings would lead a person of ordinary skill in the art to treat

the adsorbent in a packed column *and then elute* the essential oils *at a location different than the column.*

The applicants submit that the answer is "yes".

ARGUMENT

THE COMBINED TEACHINGS OF THE PRIOR ART FAIL TO TEACH AN ESSENTIAL OIL-EXTRACTION PROCESS WHEREIN THE ESSENTIAL OIL IS ELUTED, AFTER ADSORPTION, IN THE ADSORPTION COLUMN

The applicants respectfully submit that a review of the English translation of JP '699, as relied upon by the Examiner to support the § 103 rejection, reveals that the Examiner is incorrect, as JP '699 *does not* disclose that the absorption and elution take lace in the same column, and in fact *suggests that elution take place elsewhere*. That is, JP '699 suggests that elution is practiced outside of the adsorption column. Specifically, Example 1 of the English translation of JP '699 indicates that the adsorption column is packed with 6 kg of activated carbon, and states that after the washed wort vapor discharge passes through the packed column, that "10 g of activated carbon, through which has passed 5121 m^3 of vapor, [was] washed thoroughly with 200 ml of dichloromethane and filtered.[1]" The disclosure of JP '699 is clear – only 10 g of activated carbon that has adsorbed hop essential oil -- a minute fraction of the original 6 kg of activated carbon packing (0.06%, to be specific) is washed with dichloromethane and filtered. Since only a small fraction of activated carbon is subjected to elution with dichloromethane, the implication is also clear – that this small activated carbon fraction has been separated from the whole, or in other words, it has been removed from the column prior to washing and filtration. Accordingly, it appears that the examiner is

1 See also Comparative Example 1 of JP '699, found at page 7 of the English translation, which mirrors example 1 on this point.

incorrect in stating that "JP '699 teaches adsorbing into a packed column and then eluting the essential oils *from the same column*".

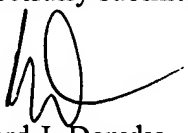
Further, in view of the teachings of JP '699, and what it fairly suggests to the skilled artisan, the applicants submit that JP '994 in view of JP '699 *teaches away* from the present invention. From the combined teachings, the skilled artisan learns to elute at a location different from where absorption takes place. Accordingly, JP '994 and JP '699 do not establish a prima facie case of nonpatentability with respect to the combined process the applicant's claimed essential oil separation process which employ the elution process specifically recited in step (d)[2].

2 The applicants recognize that Claims 1-8, 10, and 11 have been rejected under 35 U.S.C. §103(a) as allegedly unpatentable over JP (H6-227994) ("JP 994") in view of JP 60-115699 (JP 699), Chemical Engineer's Handbook, Perry and Green, 6th ed., 1984, pages 13-53 – 13-57("Perry") and Somekh (US 3,714,033) and the forgoing does not discuss Perry or Somekh. Perry or Somekh are have been cited by the Examiner to purportedly show that the step of recycling of solvent in extractive and steam distillation is "commonly used". Final office action of August 10, 2004 at page 3. The examiner has not cited any teachings of these particular references that have relevance to the elution process specifically recited in step (e) of claims 1 and 11.

CONCLUSION

Wherefore, based upon the foregoing, it is respectfully submitted that the final rejection of the claims on appeal should be reversed by the Board and that said claims are in condition of allowance.

Respectfully submitted,



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APPENDIX

1. A process for separating essential oils from an essential oil containing material comprising the steps of:

   a. subjecting the essential oil-containing material to steam distillation using a steam distillation vessel or extraction using a steam distillation vessel or extraction using an extraction vessel to produce a mixture containing the essential oil and at least one hydrophilic phase;

   b. contacting the mixture with a hydrophobic adsorbent in a column;

   c. recycling the hydrophilic phase leaving the column to the steam distillation vessel or extraction vessel; and

   d. treating the adsorbent in the column with a solvent that is more hydrophobic than the hydrophilic phase to elute the essential oil from the adsorbent.

2. A process according to Claim 1, wherein the mixture is obtained by steam distillation of the essential oil-containing material.

3. A process according to Claim 2, wherein the hydrophilic phase which is recycled in step (iii) has a temperature of 55-70°C.

4. A process to Claim 2, wherein the hydrophobic adsorbent is selected from synthetic polymers, modified silica and activated carbon.

5. A process according to Claim 4, wherein the adsorbent is selected from polystyrene, divinyl benzene-crosslinked polystyrene, and C4-, C8- and C18-coated silica.

6. A process according to Claim 1, wherein the essential oil-containing material is selected from Amber seed (=Ambrette or Musk seed), Rose, Cardamom, Cistus (=Rockrose), Costus root, Cumin, Elemi, Incence, Galbanum, Juniper, Gurjun, Lovage, Nutmeg, Orris, Myrrh, Cicely, Sweet Cicely, Styrax, Valerian, Melissa, Parsley, Bucchu, Cypress, Geranium, Lavandin, Lavender, Patchoulli, Santal (=Sandalwood), Sage, Vetyver and Ylang Ylang.

7. A process according to Claim 1, wherein the essential oil is selected from Amber oil, Ambrette, Rose oil, Cardamom oil, Cistus oil, Costus oil, Cumin oil, Elemi oil, Incence oil, Galbanum oil, Juniper oil, Gurjun oil, Lovage oil, Nutmeg oil, Orris oil, Myrrh oil, Cicely oil, Sweet Cicely oil, Styrax oil, Valerian oil, Melissa oil, Parsley oil, Bucchu oil, Cypress oil, Geranium oil, Lavandin oil, Lavender oil, Patchouli oil, Santal oil, Sage oil, Vetyver oil and Ylang Ylang oil.

8. A process according to Claim 1, wherein the treatment of step (iv) is effected as a chromatographic separation of the essential oil.

9. A process according to Claim 8, wherein the essential oil is Orris oil which is separated by the column into myristic acid and irone.

10. A process according to Claim 1, which is carried out continuously.

11. A process for separating essential oils from a plant containing an essential oil comprising the steps of:

a. subjecting the essential oil-containing plant material to steam distillation using a steam distillation vessel to produce a mixture containing the essential oil and at least one hydrophilic phase;

b. contacting the mixture with a hydrophobic adsorbent in a column;

c. recycling the hydrophilic phase leaving the column to the steam distillation vessel; and

d. treating the adsorbent in the column with a solvent that is more hydrophobic than the hydrophilic phase to elute the essential oil from the adsorbent.